

# **U. S. Department of Energy**



## **Consolidated Audit Program**

### **Module 3**

### **Checklist for Inorganics**

**Revision 2  
February 17, 2004**

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| Item  | Line of Inquiry  | Status | Summary of Observations/Objective Evidence Reviewed/Audit Notes |
|-------|--|--------|---|
| 3.1   | Standard Operating Procedures  |        |   |
| 3.1.1 | <p>The laboratory maintains and follows Standard Operating Procedures (SOPs) for each inorganic preparation and analytical method performed at their facility. These SOPs meet the following criteria:</p> <ul style="list-style-type: none"> <li>complete and correct;</li> <li>contain sufficient information to perform the specified analysis; and,</li> <li>readily available to analysts.</li> </ul> <p><i>(Quality Systems for Analytical Services, 10.1.1)</i></p> |        |   |
| 3.1.2 | <p>Analytical procedures followed by the analyst agree with the written SOPs .</p> <p><i>(Quality Systems for Analytical Services, 10.1.1e)</i></p>  |        |   |
| 3.1.3 | <p>A system is in place to ensure that quality records are legible, accurate, and complete. e.g., independent review of records, logbooks, etc.</p> <p><i>(SW-846, Chapter One, Section 4.6, Quality Systems for Analytical Services, 12.1)</i></p>  |        |   |

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| 3.1.4 | <p>All observations and results recorded by the laboratory are on pre-printed forms, electronic media, or entered into permanent laboratory logbooks.</p> <p><i>(Quality Systems for Analytical Services, 12.0)</i></p>  |        |   |
| 3.1.5 | <p>Laboratory notebooks (logbooks) comply with the following:</p> <ul style="list-style-type: none"> <li>are controlled through a documented system;</li> <li>have sequentially numbered pages;</li> <li>have unique serial number clearly displayed on each notebook;</li> <li>entries made in permanent fashion and corrections made without obliterating original entries;</li> <li>entries dated and signed by the person responsible for performing the activity at the time the activity is performed; and,</li> <li>entries are in chronological order.</li> </ul> <p><i>(Quality Systems for Analytical Services, 12.2d)</i></p> |        |   |
| 3.1.6 | <p>Corrections to documents that will become quality records are made by drawing a single line through the error, initialing the error, and justifying the correction, if non self-explanatory.</p> <p><i>(Quality Systems for Analytical Services, 12.1f)</i></p>   |        |   |

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| Item  | Line of Inquiry  | Status | Summary of Observations/Objective Evidence Reviewed/Audit Notes |
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| 3.2   | <b>Balances</b>  |        |   |
| 3.2.1 | Balances are located in an area where the environment has little or no effect on measurement accuracy.<br><br>( <i>Quality Systems for Analytical Services, 7.1a</i> )   |        |   |
| 3.2.2 | Balances are checked each day that they are used and are calibrated at least annually by an independent company or source.<br><br>( <i>Quality Systems for Analytical Services, 9.4.1b and d</i> )   |        |   |
| 3.2.3 | Check weightings are performed daily at three points within the balance range using NIST traceable weights. Daily checks are documented in controlled logbooks, on-preprinted forms or electronically.<br><br>( <i>Quality Systems for Analytical Services, 9.4.1b and d</i> ) |        |   |
| 3.2.4 | Balance check weights shall bracket the range of use (sample weight).<br><br>( <i>Quality Systems for Analytical Services, 9.4.1 b and d</i> )   |        |   |

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| <b>3.3</b> | <b>Thermometers and Pipettes</b>  |  |  |
| 3.3.1      | Liquid-in-glass thermometers are calibrated against a NIST traceable standard at least every five years.<br><br><i>(Quality Systems for Analytical Services, 9.4.1 b)</i>   |  |  |
| 3.3.2      | Thermometers are uniquely identified by an identification protocol.<br><br><i>(Quality Systems for Analytical Services, 8.0d)</i>   |  |  |
| 3.3.3      | The accuracy of all non-Class A pipettes and automatic sample dispensers used for quantitative measurement is verified monthly or whenever degradation of measuring equipment is suspected.<br><br><i>(Quality Systems for Analytical Services, 9.4.1e)</i> |  |  |
| 3.3.4      | Pipettes and automatic sample dispensers are uniquely identified by an identification protocol.<br><br><i>(Quality Systems for Analytical Services, 8.0d)</i>   |  |  |

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| <b>3.4</b> | <b>Glassware</b>  |  |  |
| 3.4.1      | The laboratory-specific SOP for glassware shall be available to applicable laboratory personnel.<br><br><i>(Quality Systems for Analytical Services, 7.2c)</i>                        |  |  |
| 3.4.2      | Sample glassware and containers are either designated as disposable or cleaned according to a SOP.<br><br><i>(Quality Systems for Analytical Services, 9.4.1e and Appendix D.1.8)</i> |  |  |
| <b>3.5</b> | <b>Sample Preparation</b>   |  |  |
| 3.5.1      | Sample preparation areas are kept clean to avoid contamination.<br><br><i>(Quality Systems for Analytical Services, 7.2c and d)</i>   |  |  |
| 3.5.2      | All sample preparations are conducted in a hood.<br><br><i>(Quality Systems for Analytical Services, 7.2)</i>   |  |  |

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| <b>3.6</b> | <b>Metals Digestion</b>  |  |  |
| 3.6.1      | <p>The temperature of metals digestion equipment (hotplate, hot block, water baths, etc.) is monitored and recorded on a regular basis. Corrective action is taken if the temperature of the metals digestion equipment falls outside the range established by the laboratory.</p> <p><i>(Quality Systems for Analytical Services, 9.4.1d)</i></p> |  |  |
| 3.6.2      | <p>The laboratory uses water meeting ASTM specifications (or equivalent) for “Type II” water (ASTM D1193) for digestion procedures.</p> <p><i>(SW-846 Chapter One, Section 5.0 Definitions – Reagent Water)</i></p>  |  |  |
| 3.6.3      | <p>For atomic spectroscopy, ASTM Type I water used for standards preparation, blanks, and sample dilutions. (max conductivity = 0.06 <math>\mu</math>ohms/cm, min resistivity = 16.67 megohms-cm)</p> <p><i>(SW-846 Method 7000A Section 5.2)</i></p>  |  |  |
| 3.6.4      | <p>The laboratory uses reagent or trace metal grade acid for digestion procedures.</p> <p><i>(SW-846 Method specific Section 5.0 and Quality Systems for Analytical Services, Appendix D-1.4)</i></p>  |  |  |

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| 3.6.5      | For microwave digestion, the laboratory employs an oven with programmable power settings. A procedure is in place for the operation of the oven.<br><br><i>(Quality Systems for Analytical Services, 10.1a)</i>  |  |  |
| 3.6.6      | The drying oven: <ul style="list-style-type: none"> <li>• has a temperature measurement device; and,</li> <li>• the temperature is monitored and documented.</li> </ul> <i>(Quality Systems for Analytical Services 9.4.1d)</i>  |  |  |
| 3.6.7      | Method blanks, spiked samples, and laboratory control samples are carried through the same digestion process.<br><br><i>(Quality Systems for Analytical Services, Appendix D-1.1)</i>  |  |  |
| <b>3.7</b> | <b>Method Detection Limits</b>   |  |  |
| 3.7.1      | A procedure is in place for determination and documentation of method detection limits for each inorganic method performed by the laboratory. The laboratory maintains procedures for determining for limits of detection and the frequency of verification.<br><br><i>(Quality Systems for Analytical Services, 5.4a)</i> |  |  |



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| 3.7.2      | Method detection limits have been established by analytical method for each instrument configuration and matrix. MDLs shall be determined by the protocol in the mandated test method.<br><br><i>(Quality Systems for Analytical Services, Appendix D.1.4)</i>   |  |  |
| 3.7.3      | The laboratory maintains documentation for each method detection limit study.<br><br><i>(Quality Systems for Analytical Services, Appendix D-1.2)</i>  |  |  |
| <b>3.8</b> | <b>Analytical Standards and Reagents</b>   |  |  |
| 3.8.1      | Standards and reference materials are traceable to EPA or NIST certified standards, including:<br><br><ul style="list-style-type: none"> <li>• initial calibration standards;</li> <li>• continuing calibration standards; and</li> <li>• spiking standards.</li> </ul> <i>(Quality Systems for Analytical Services, Appendix D.1.1)</i> |  |  |

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| 3.8.2 | Standards are assigned a unique identification number traceable to the original standard. The unique identification number and the expiration date are placed on the standards container.<br><br><i>(Quality Systems for Analytical Services, 10.5d)</i>  |  |  |
| 3.8.3 | Labels for purchased stock mixtures and reagents contain the following information:<br><br><ul style="list-style-type: none"> <li>• date received;</li> <li>• date opened; and</li> <li>• expiration date.</li> </ul> <i>(Quality Systems for Analytical Services, 10.5 a and b)</i>  |  |  |
| 3.8.4 | Secondary standard solutions are traceable to a standards preparation log and labeled with the following information:<br><br><ul style="list-style-type: none"> <li>• secondary standard tracking identification number;</li> <li>• preparer's initials;</li> <li>• preparation date; and</li> <li>• secondary standard expiration date.</li> </ul> <i>(Quality Systems for Analytical Services, Section 10.5c and d)</i> |  |  |

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| 3.8.5 | <p>Standards and reference materials logbook of standards preparation contains the following information:</p> <ul style="list-style-type: none"> <li>• standards ID number;</li> <li>• standards prepared;</li> <li>• matrix noted;</li> <li>• spiking standards;</li> <li>• pretreatment;</li> <li>• volume/weight of Standards;</li> <li>• final volume; and,</li> <li>• preparation methods.</li> </ul> <p><i>(Quality Systems for Analytical Services, 10.5c)</i></p> |  |  |
| 3.8.6 | <p>Standards and reference materials are stored separately from samples and standards are protected in a controlled cabinet or refrigerator.</p> <p><i>(Quality Systems for Analytical Services, 10.5a)</i></p>   |  |  |
| 3.8.7 | <p>Calibration standards are prepared using certified standards traceable to a nationally recognized or consensus source (i.e., NIST or equivalent SRM) and the certificates of authenticity are kept on file.</p> <p><i>(Quality Systems for Analytical Services, 9.2)</i></p>   |  |  |

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| 3.8.8      | <p>If the initial calibration is not verified using an independent source, the LCS is prepared from an independent source.</p> <p><i>(Quality Systems for Analytical Services, 3.0 LCS)</i></p>   |  |  |
| 3.8.9      | <p>The laboratory has a procedure in place to track the expiration date of standards and removes standards from use when expired.</p> <p><i>(Quality Systems for Analytical Services, 10.5a)</i></p>  |  |  |
| <b>3.9</b> | <b>Instrument Operations and Maintenance</b>  |  |  |
| 3.9.1      | <p>The laboratory maintains a current list of equipment types, models and year of manufacture.</p> <p><i>(Quality Systems for Analytical Services, 8.0c)</i></p>  |  |  |
| 3.9.2      | <p>A system is in place to address instrument operational problems. This system should include the following elements;</p> <ul style="list-style-type: none"> <li>• fault finding/troubleshooting;</li> <li>• procedures to repair malfunctioning equipment; and,</li> <li>• actions to be taken to prevent recurrence.</li> </ul> <p><i>(Quality Systems for analytical Services, 10.1.1e)</i></p> |  |  |

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| 3.9.3 | The laboratory maintains an in-house inventory of replacement parts for equipment and instruments or has a service contract for instrumentation.<br><br><i>(Quality Systems for Analytical Services, 8.0c)</i>                                 |  |  |
| 3.9.4 | The laboratory has established maintenance procedures and schedule for routine instrument maintenance.<br><br><i>(Quality Systems for Analytical Services, 8.0b and 8.0e)</i>  |  |  |
| 3.9.5 | Records are maintained for maintenance performed on analytical instrumentation. Instrument modifications are documented in a permanent record.<br><br><i>(Quality Systems for Analytical Services, 8.0e)</i>                                   |  |  |
| 3.9.6 | Out-of-calibration equipment is tagged or segregated and not used until it has been re-calibrated. Equipment consistently found to be out of calibration is repaired or replaced.<br><br><i>(Quality Systems for Analytical Services, 8.0)</i> |  |  |
| 3.9.7 | The laboratory has a procedure in place to notify laboratory personnel when equipment is taken out of service.<br><br><i>(Quality Systems for Analytical Services, 10.1.1)</i>   |  |  |

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| 3.9.8       | Analysts have a solid understanding of analytical anomalies and the instrument conditions that cause them.<br><br><i>(Quality Systems for Analytical Services, 6.0)</i>  |  |  |
| 3.9.9       | Calibration results are maintained as a permanent laboratory record and contain sufficient information to allow monitoring of instrument performance over time.<br><br><i>(Quality Systems for Analytical Services, 9.4.2.1b and 9.4.2.2c)</i> |  |  |
| 3.9.10      | The laboratory has a procedure in place to determine and correct elemental interferences.<br><br><i>(Quality Systems for Analytical Services, 10.1.1)</i>  |  |  |
| <b>3.10</b> | <b>Method Blanks</b>   |  |  |
| 3.10.1      | Method blanks are prepared and analyzed with each batch of 20 samples or less.<br><br><i>(Quality Systems for Analytical Services, Appendix D-1.1a)</i>  |  |  |

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| 3.10.2      | When a method blank fails to meet the laboratory or method acceptance criteria, a corrective action process is initiated.<br><br><i>(Quality Systems for Analytical Services, Appendix D-1.1a)</i>  |  |  |
| <b>3.11</b> | <b>Laboratory Control Sample</b>  |  |  |
| 3.11.1      | A laboratory control sample is prepared and analyzed with each batch of 20 samples or less.<br><br><i>(Quality Systems for Analytical Services, Appendix D-1.b(1))</i>  |  |  |
| 3.11.2      | The results of the LCS shall be calculated in percent recovery and the calculation shall be documented. The laboratory shall define acceptance limits. When a laboratory control sample fails to meet the laboratory or method acceptance criteria, a corrective action process is initiated.<br><br><i>(Quality Systems for Analytical Services, Appendix D-1.1b(1))</i> |  |  |
| <b>3.12</b> | <b>Matrix Spike/Matrix Spike Duplicate</b>  |  |  |
| 3.12.1      | One Matrix Spike/Matrix Spike Duplicate (MS/MSD) or matrix spike/duplicate is prepared and analyzed with  |  |  |

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|             | each batch of 20 samples or less.<br><br><i>(Quality Systems for Analytical Services, Appendix D-1.1c)</i>  |  |  |
| 3.12.2      | When a MS/MSD or matrix spike/duplicate fails to meet the laboratory or method acceptance criteria, a corrective action process is initiated.<br><br><i>(Quality Systems for Analytical Services, Appendix D1-1c)</i>                       |  |  |
| <b>3.13</b> | <b>Multipoint Calibration Procedures - Graphite Furnace Atomic Absorption (GFAA) SW-846, Method 7000A</b>   |  |  |
| 3.13.1      | For each instrument in use, a calibration procedure is performed daily consisting of three standards of appropriate concentrations such that an effective linear range can be established.<br><br><i>(SW-846 Method 7000A, Section 8.2)</i> |  |  |
| 3.13.2      | The calibration standards are prepared fresh at the time of analysis.<br><br><i>(SW-846 Method 7000A, Section 8.2)</i>  |  |  |
| 3.13.3      | The calibration curve is verified using a calibration blank and a calibration check standard derived from a source independent of that used to prepare the initial calibration standards.   |  |  |



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|        | (SW-846 Method 7000A, Section 8.2)   |  |  |
| 3.13.4 | When more than 10 samples are analyzed on a particular instrument per day, the multipoint calibration is verified after every 10 samples by analyzing a standard at the mid-range of the curve.<br><br>(SW-846 Method 7000A, Section 8.3)  |  |  |
| 3.13.5 | The analyst documents the addition of any matrix modifiers to the sample.<br><br>(Quality Systems for Analytical Services, 12.3.3g)  |  |  |
| 3.13.6 | <u>Dilution Test</u> : One diluted sample is analyzed per batch. The concentration of the analyte should be at least 25 times the estimated detection limit. Dilute the sample by at least 5 fold. Concentrations between the undiluted and diluted sample agree within 10% if there are not interferences.<br><br>(SW-846 Method 7000A Section 8.6) |  |  |
| 3.13.7 | <u>Recovery Test</u> : If interferences exist or if all the samples in the batch are <10 times the MDL perform a recovery test. Add a known amount of the analyte to bring the concentration of the analyte to 2 to 5 times the original concentration. The spike recovery should be   |  |  |

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|             | 85-115%.<br><br>(SW-846 Method 7000A, Section 8.6.2)  |  |  |
| 3.13.8      | <p><u>Method of Standard Addition (MSA)</u>. If the spike recovery test does not yield a recovery of 85-115%, the method of standard addition is applied to all samples in the batch.</p> <p>The laboratory employs the MSA when interference is suspected or a new matrix is encountered.</p> <p>(SW-846, Method 7000A, Section 8.7)</p>   |  |  |
| <b>3.14</b> | <b>Inductively Coupled Plasma Mass Spectrometry (ICP-MS) SW-846 Method 6020</b>   |  |  |
| 3.14.1      | <p><u>Instrument Detection Limits (IDLs)</u>: IDLs are established by calculating the average of the standard deviations of three runs on three-non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Each measurement shall be performed as a separate analytical sample. IDLs shall be determined at least every three months and kept with the instrument log book.</p> <p>(SW-846 Method 6020, Section 8.2)</p> |  |  |
| 3.14.2      | <p>Mass calibration and resolutions checks are conducted in the mass regions of interest.</p> <p>(SW-846, Method 6020, Section 7.5)</p>   |  |  |

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| 3.14.3 | <p><u>Internal Standards:</u> Laboratory procedures specify the selection and use of internal standards and their associated acceptance criteria.</p> <p><i>(Quality Systems for Analytical Services, 10.1.1)</i></p>   |  |  |
| 3.14.4 | <p>For each instrument in use, a calibration procedure is performed for the analytes of interest consisting of (at a minimum) a blank and a standard. The average of at least three integrations is used for calibration.</p> <p><i>(SW-846 Method 6020, Section 7.6)</i></p> |  |  |
| 3.14.5 | <p>A calibration blank and either a CCV or an ICV are analyzed after every tenth sample and at the end of the sample run.</p> <p><i>(SW-846 Method 6010B, Section 7.4)</i></p>  |  |  |
| 3.14.6 | <p>The calibration is verified for every analyte by the analysis of a calibration verification solution.</p> <p><i>(SW-846 Method 6020, Section 7.8)</i></p>  |  |  |
| 3.14.7 | <p>A corrective action process is implemented when the calibration check standards are not within 10% of the control limit established by the laboratory.</p> <p><i>(SW-846 Method 6020, Section 7.8)</i></p>   |  |  |

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| 3.14.8  | Interference levels are corrected by the instrument data system and are accounted for in the data report.<br><br><i>(SW-846 Method 6020, Section 4.1)</i>  |  |  |
| 3.14.9  | The interference check solution (ICS) is prepared to contain known concentrations of interfering elements that will demonstrate the magnitude of interferences and provide an adequate test of any corrections.<br><br><i>(SW-846 Method 6020, Section 8.9)</i>  |  |  |
| 3.14.10 | ICSs are analyzed at the beginning of an analytical run or once every 12 hours, whichever is more frequent.<br><br><i>(SW-846 Method 6020, Section 8.9)</i>  |  |  |
| 3.14.11 | <u>Post Digestion Spike</u> : An analyte spike is added to a portion of a prepared sample. If the recovery is not within 85-115%, the dilution test or MSA is performed. Results of the dilution should be +/-10% of the original sample concentration. The spike is prepared at a concentration to yield a result between 10 and 100 times the IDL.<br><br><i>(SW-846 Method 6020, Section 8.6)</i> |  |  |

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| 3.14.12     | <p><u>Dilution Test</u>: For each new matrix, a dilution test is performed. Analyze a five-fold dilution on a sample containing analytes &gt;100 times the reagent blank. Results of the dilution must agree with +/-10% of the original determination.</p> <p><i>(SW-846 Method 6020, Section 8.5)</i></p>               |  |  |
| <b>3.15</b> | <b>Multipoint Calibration Procedures – Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES) Method 6010B</b>   |  |  |
| 3.15.1      | <p><u>Linear Dynamic Range (LDR)</u>: Determine the signal responses from a minimum of three different concentration standards across the range. The upper range limit should be within +/-10% of the highest measured level. LDR are verified every six months.</p> <p><i>(SW-846 Method 6010B, Section 7.2.5.4)</i></p> |  |  |
| 3.15.2      | <p><u>Interelement Correction Factor (IEC) Factor</u>: IEC factors must be determined and verified every six months. Data for interelement correction factors is maintained in a manner that is readily accessible to the analyst.</p> <p><i>(SW-846 Method 6010B, Sections 3.1 and 7.2.3.6)</i></p>                      |  |  |

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| 3.15.3 | For each instrument in use, a calibration procedure is performed daily consisting of (at a minimum) a blank and standard.<br><br><i>(SW-846 Method 6010B, Section 7.3)</i>  |  |  |
| 3.15.4 | Following daily calibration, an Initial Calibration Verification (ICV), calibration blank, and Continuing Calibration Verification (CCV) are analyzed.<br><br><i>(SW-846 Method 6010B, Sec. 7.4 and Quality Systems for Analytical Services, 9.4.2.2)</i> |  |  |
| 3.15.5 | A calibration blank and either CCV or an ICV are analyzed after every tenth sample and at the end of the sample run.<br><br><i>(SW-Method 6010B, Section 7.4)</i>   |  |  |
| 3.15.6 | The results of the calibration blank are within three times the IDL. If not, a corrective action is initiated.<br><br><i>(SW-846 Method 6010B, Section 8.6.1.3)</i>   |  |  |
| 3.15.7 | When the calibration verification falls outside the method or laboratory established criteria, a corrective action process is initiated.<br><br><i>(SW-846 Method 6010B, Section 7.4)</i>   |  |  |

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| 3.15.8  | <p><u>Interference Check Solutions (ICS)</u>: ICSs are analyzed at the beginning of an analytical run or once every 12 hours, whichever is more frequent.</p> <p><i>(SW-846 Method 6010B, Section 8.6.2)</i></p>  |  |  |
| 3.15.9  | <p><u>Dilution Test</u>: For each new matrix, a dilution test is performed. Analyze a five-fold dilution on a sample containing analytes &gt;100 times the reagent blank. Results of the dilution must agree within +/-10% of the original determination.</p> <p><i>(SW-846 Method 6010B, Section 8.5.1)</i></p>  |  |  |
| 3.15.10 | <p><u>Post Digestion Spike</u>: For each new matrix, an analyte spike is added to a portion of a prepared sample. If the recovery is not within 85-115%, the dilution test or MSA is performed. Results of the dilution should be +/- 10% of the original sample concentration. The spike is prepared at a concentration to yield a result between 10 and 100 times the IDL.</p> <p><i>(SW-846 Method 6010B, Section 8.5.2)</i></p> |  |  |

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| <b>3.16</b> | <b>Multipoint Calibration Procedures - Cold Vapor Atomic Absorption Spectrometry (CVAA) Methods: Hg Analysis by SW-846 Methods 7470A and 7471A</b>             |  |  |
| 3.16.1      | The multipoint calibration is performed using 5 standards and a blank.<br><br><i>(SW-846 Method 7470A Section 7.0 and Method 7471A Section 7.1)</i>            |  |  |
| 3.16.2      | The working standards are prepared fresh before the analysis.<br><br><i>(SW-846 Method 7470A Section 5.10 and Method 7471A Section 5.8)</i>                    |  |  |
| 3.16.3      | If the laboratory performs EPA Method 7471A, aqua regia is used for sample and standards preparation.<br><br><i>(SW-846 Method 7471A Sections 7.1 and 7.3)</i> |  |  |



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| <b>3.17</b> | <b>Quality Control of Instrument Gases</b>  |  |  |
| 3.17.1      | <p>Fuel and oxidant gases of the appropriate quality to insure proper instrument performance are used, including, but not limited to, the following:</p> <ul style="list-style-type: none"> <li>• acetylene (high purity);</li> <li>• compressed air (“clean and dry”);</li> <li>• nitrous oxide;</li> <li>• argon (commercial grade); and,</li> <li>• nitrogen (commercial grade).</li> </ul> <p><i>(SW-846 Method 7000A, Section 5.5)</i></p> |  |  |
| <b>3.18</b> | <b>Wet Chemistry Procedures Cyanide Distillation by SW-846 Method 9010 and Analysis by Methods 9014</b>   |  |  |
| 3.18.1      | <p>The laboratory uses 6 standards and a blank for the multipoint calibration.</p> <p><i>(SW-846 Methods 9014 Sections 7.3 and 7.4)</i></p>   |  |  |
| 3.18.2      | <p>The laboratory distills and analyzes high and low standards. The results are within 10% of the un-distilled results.</p> <p><i>(SW-846 Method 9010B Section 9.6)</i></p>   |  |  |

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| 3.18.3      | At least one matrix spike sample is analyzed with each batch of 20 samples or less through the entire sample preparation and analytical process.<br><br>(SW-846 Method 9010B Section 8.5)                 |  |  |
| 3.18.4      | One replicate sample is analyzed with each batch of 20 samples or less.<br><br>(SW-846 Method 9010B Section 8.4)  |  |  |
| <b>3.19</b> | <b>Reactivity Determinations - Colorimetric Cyanide by SW-846 Method 9012A</b>  |  |  |
| 3.19.1      | Calibration curves include: <ul style="list-style-type: none"> <li>a minimum of five points, including a blank; and,</li> <li>a standard near the midpoint.</li> </ul> (SW-846 Method 9012 Section 7.4.1) |  |  |
| <b>3.20</b> | <b>Reactivity Determinations - Titrimetric Sulfide</b>  |  |  |
| 3.20.1      | Titration concentration values are traceable to primary standards.  |  |  |
| 3.20.2      | Each analytical batch includes at least one titration blank.  |  |  |

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| 3.21   | TCLP Extractions – SW-846 Method 1311 Toxicity Characteristic Leaching Procedure (TCLP)   |  |  |
| 3.21.1 | Agitation apparatus rotates at 30 +/- 2 rpm. Verification of the rotation is documented and a frequency of the verification has been defined.<br><br>(SW-846 Method 1311 Section 4.1)   |  |  |
| 3.21.2 | Zero Headspace Extraction (ZHE) units are leak checked after every extraction by monitoring the pressure gauge or by pressurizing the unit, submerging in water and checking for bubbles. The laboratory has a corrective action procedure for handling leaking ZHE vessels.<br><br>(SW-846 Method 1311 Section 4.2.1)                          |  |  |
| 3.21.3 | <u>ZHE Extract Collection Devices</u> : The final extract for ZHE is collected in Tedlar bags or glass using stainless steel or PTFE gas-right syringes.<br><br>(SW-846 Method 1311 Section 4.6)  |  |  |
| 3.21.4 | <u>Extraction Fluid Reagents</u> : Extraction fluid reagents are prepared using ASTM Type II water and reagent grade chemicals or equivalent. The pH of extraction fluid 1 is 4.93 +/- 0.05 (4.88-4.98) and the pH of extraction fluid 2 2.88 +/-0.05 (2.83-2.93). The pH must be checked prior to use.<br><br>(SW-846 Method 1311 Section 5.7) |  |  |

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| 3.21.5 | <p><u>Determination of Percent Solids:</u> If the percent solid is determined to be &lt;0.5%, the sample is filtered through 0.6-0.8µm filter paper: solids are discarded and the liquid is analyzed. If the percent solid is determined to be &gt;0.05% the sample is filtered through 0.6-0.8µm filter paper, liquids are separated and solids and extract solids.</p> <p><i>(SW-846 Method 1311, Section 7.1.1)</i></p> |  |  |
| 3.21.6 | <p><u>Determination of Particle Size Reduction:</u> Particle size should be &lt;9.5mm.</p> <p><i>(SW-846 Method 1311 Section 7.1.3)</i></p>  |  |  |
| 3.21.7 | <p><u>Determination of Appropriate Extraction Fluid:</u> For ZHE extraction, fluid #1 is used. For TCLP extraction, determine fluid based on pH. pH&lt;5 use fluid #1; pH&gt;5 even after addition of HCL use fluid #2</p> <p><i>(SW-846 Method 1311 Section 7.1.4)</i></p>  |  |  |
| 3.21.8 | <p>The room temperature is maintained at 23 +/- 2°C during agitation.</p> <p><i>(SW-846 Method 1311 Sections 7.2.10 and 7.3.12.3)</i></p>  |  |  |

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| 3.21.9  | TCLP and ZHE extraction devices are rotated at 30+/-2 rpm for 18 +/- 2 hours.<br><br><i>(SW-846 Method 1311 Sections 7.2.10 and 7.3.12.3)</i>   |  |  |
| 3.21.10 | Immediately following TCLP extraction collection, the pH of the extract is recorded and the extract is preserved for analysis. Metals aliquots must be acidified with nitric acid to pH<2 and organic aliquots must be refrigerated.<br><br><i>(SW-846 Method 1311, Section 7.2.14)</i> |  |  |
| 3.21.11 | The method blank for TCLP/ZHE must be of the same leaching fluid as the sample(s), tumbled in an extraction vessel and filtered in the same manner as the sample(s).<br><br><i>(SW-846 Method 1311 Section 8.1)</i>   |  |  |
| 3.21.12 | A minimum of one matrix spike shall be performed for each batch. Matrix spikes are added after filtration of the TCLP/ZHE extract and prior to preservation.<br><br><i>(SW-846 Method 1311 Section 8.2)</i>   |  |  |

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| 3.22   | <b>Ignitability Test: SW-846 Methods 1010 Pensky-Martin Closed Cup and Method 1020A Seta-Flash Closed Cup or ASTM D93.94 Pensky-Martens or ASTM D3278-82 Seta-flash</b> |  |  |
| 3.22.1 | The stirring and heat up rates are monitored and documented.  |  |  |
| 3.22.2 | Thermometers have been calibrated for measuring the flash point temperature.  |  |  |
| 3.22.3 | The apparatus employed by the laboratory meets the requirements of either ASTM D93-94 (Pensky-Martens) or ASTM D3278-82 (Seta-Flash).                                   |  |  |
| 3.22.4 | When samples contain non-filterable suspended solids or liquids that form surface films, the Pensky-Martens method is used.<br><br>(SW-846 Method 1020A Section 1.3)    |  |  |
| 3.22.5 | Calibrations are verified using a fluid with a known flash point.   |  |  |
| 3.22.6 | The calibration verification is performed immediately after calibration, after each series of 10 determinations and after the last site sample has been analyzed.       |  |  |

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| 3.22.7      | Two measurements are obtained for each sample. A corrective action process is initiated if duplicate measurement results are not within established limits.<br><br>(SW-846 Method 1020A Section 2.3)  |  |  |
| <b>3.23</b> | <b>Corrosivity Determination: pH Electrometric Measurement by SW-846 Method 9040B</b>   |  |  |
| 3.23.1      | pH meters in operation at the time of the audit have the following characteristics: <ul style="list-style-type: none"> <li>• temperature compensating;</li> <li>• read to 2 decimal places; and,</li> <li>• capable of measuring sample and reference standards temperatures to 0.1°C.</li> </ul> (SW-846 Method 9040B)                             |  |  |
| 3.23.2      | Apparatus includes a magnetic stirrer with stirring bar made of non-reactive material such as Teflon.<br><br>(SW-846 Method 9040B Section 4.4)  |  |  |
| 3.23.3      | The following calibration criteria are met: <ul style="list-style-type: none"> <li>• performed daily and each time the instrument is set up;</li> <li>• curve contains a minimum of two points that are at least 2.8 pH units apart;</li> <li>• verification performed immediately after calibration, each 10 samples and after the last</li> </ul> |  |  |

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| 3.23.3<br>Cont'd | <p>site sample is analyzed; and,</p> <ul style="list-style-type: none"> <li>• verification near the midpoint of the calibration line.</li> </ul> <p><i>(SW-846 Method 9040B Section 7.1)</i></p>   |  |  |
| <b>3.24</b>      | <b>Corrosivity Towards Steel by SW-846 Method 1110A</b>  |  |  |
| 3.24.1           | <p>Test apparatus includes:</p> <ul style="list-style-type: none"> <li>• kettle or flask;</li> <li>• reflux condenser;</li> <li>• thermowell;</li> <li>• temperature regulating device;</li> <li>• heating device; and,</li> <li>• specimen support system.</li> </ul> <p><i>(SW-846 Method 1110A Section 4.1)</i></p> |  |  |
| 3.24.2           | <p>Test specimen is made of SAE1020 steel.</p> <p><i>(SW-846 Method 1110A Section 4.5)</i></p>   |  |  |
| 3.24.3           | <p>Specimen surface area measurement is accurate to +/-1%</p> <p><i>(SW-846 Method 1110A Section 4.5.1)</i></p>  |  |  |



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| 3.24.4      | Duplicate samples should be analyzed on a routine basis.<br><br>(SW-846 Method 1110A Section 4.1)   |  |  |
| <b>3.25</b> | <b>Total Organic Carbon (TOC) by EPA Method 9060A</b>   |  |  |
| 3.25.1      | Samples are stored at 4 ± 2°C.<br><br>(SW-846 Method 9060 Section 6.3)  |  |  |
| 3.25.2      | The calibration of the instrument is verified by an independently prepared check standard every 15 samples.<br><br>(SW-846 Method 9060 Section 8.3) |  |  |
| 3.25.3      | Blanks are processed and analyzed with each batch of 20 samples or less.<br><br>(SW-846 Method 9060 Section 8.2)                                    |  |  |
| 3.25.4      | One spike duplicate sample shall be analyzed for every 10 samples.<br><br>(SW-846 Method 9060 Section 8.4)  |  |  |

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| 3.25.5      | Samples are analyzed in quadruplicate as required by the method. Both the average and the range are reported.<br><br>(SW-846 Method 9060 Section 7.6)   |  |  |
| <b>3.26</b> | <b>Total Organic Halogens (TOX) by SW-846 Methods 9020B and 9022</b>  |  |  |
| 3.26.1      | Samples are analyzed in duplicate.<br><br>(SW-846 Method 9020B Section 8.2)   |  |  |
| 3.26.2      | At least 2 blanks are analyzed to establish the repeatability of the method background prior to sample analysis. Method blanks are analyzed between every 8 analytical determinations.<br><br>(SW-846 Method 9020B Section 8.3) |  |  |
| <b>3.27</b> | <b>COD Determinations EPA Method 405.1 and Standard Methods 5210B</b>   |  |  |
| 3.27.1      | The dissolved oxygen meter is calibrated before and after use.  |  |  |

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| 3.28   | <b>Total Phosphorus, Nitrate/Nitrite and Anions Determinations: SW-846 Method 9056 Determination of Inorganic Anions by Ion Chromatography (chloride, fluoride, bromide, nitrate, nitrite, phosphate and sulfate)</b> |  |  |
| 3.28.1 | All reported results were made within the calibration curve of the instrument.<br><br><i>(Quality Systems for Analytical Services, 9.4.2 and SW-846 Method 9056 Section 7.2.2.10)</i>                                 |  |  |
| 3.28.2 | Calibration curve consists of at a minimum 3 concentration levels for each analyte of interest and a blank.<br><br><i>(SW-846 Method 9056 Section 7.1.2)</i>  |  |  |
| 3.28.3 | A midrange calibration standard is analyzed after every 10 injections. The instrument is recalibrated if the response changes more than 5%.<br><br><i>(SW-846 Method 9056 Section 8.2)</i>                            |  |  |
| 3.28.4 | A duplicate sample is analyzed with every ten samples.<br><br><i>(SW-846 Method 9056 Section 8.3)</i>   |  |  |
| 3.28.5 | A linear calibration plot with an acceptable correlation coefficient, intercept and slope is included with the raw data package.<br><br><i>(SW-846 Method 9056 Section 7.3.3)</i>                                     |  |  |

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| 3.28.6      | The samples are filtered or allowed to settle prior to analysis. Samples that contain particles larger than 0.45µm and reagent solutions that contain particles larger than 0.20µm require filtration to prevent damage to instrument columns and flow systems.<br><br>(SW-846 Method 9056 Section 3.4) |  |  |
| 3.28.7      | The retention time for each analyte is within 10% of the calibration standards average retention time ( <i>anion analysis only</i> ). Retention times are documented.<br><br>(Quality Systems for Analytical Services, Appendix D.1.7 and SW-846 Method 9056 Section 7.1.4)                             |  |  |
| <b>3.29</b> | <b>Sample Dilutions</b>   |  |  |
| 3.29.1      | Samples with concentrations that exceed the calibration range must be diluted to fall within the range.<br><br>(Quality Systems for Analytical Services, 9.4.2.1f)  |  |  |
| 3.29.2      | Samples initially diluted that have no identified compounds are re-analyzed at a lower dilution.<br><br>(Quality Systems for Analytical Services, Section 9.4.2.1f)   |  |  |

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| <b>3.30</b> | <b>Data Review</b>   |  |  |
| 3.30.3      | A data review procedure is in place that includes both analyst and independent data reviewer.<br><br><i>(Quality Systems for Analytical Services, 10.4b)</i>   |  |  |
| 3.30.4      | Data review is documented and records are maintained.<br><br><i>(Quality Systems for Analytical Services, 12.3.2h)</i>   |  |  |
| 3.30.5      | If manual transcriptions or data entry occur, these steps are identified and special controls are in place to check for human errors.<br><br><i>(Quality Systems for Analytical Services, 12.3.3e)</i> |  |  |
| 3.30.4      | Spreadsheets used for calculations shall be verified before use and documentation shall be readily available for review.<br><br><i>(Quality Systems for Analytical Services, 10.6c)</i>                |  |  |